

## TITLE OF THE INVENTION

### CONTINUOUS CHROMATE-FREE FLUIDIZED-BED PIPE COATING

## BACKGROUND OF THE INVENTION

### 5 FIELD OF THE INVENTION

The invention relates to a process which permits the continuous chromium-free coating of pipe.

### DESCRIPTION OF RELATED ART

Coated pipes for the automotive industry have hitherto been produced by  
10 coating processes that include the use of chromium VI compounds (chromates).  
Chromates are used in order to achieve very good adhesion values in the extrusion  
process employed to manufacture such pipes. Chromate-treated pipes as well as  
chromate-treated aluminum pipes and steel pipes that are first aluminum-treated  
and then chromate-treated have been manufactured in this manner. However, the  
15 automotive industry recently began a switch to chromium-free pipes.

Processes for the continuous coating of pipes are described in, for example,  
the journal "Kunststoffe", Volume 57, No. 1, pages 21-24, which discloses a  
process which uses fluidized-bed coating to coat pipes with PVC. This process is  
not disclosed to provide good adhesion values or layers of homogeneous thickness.  
20 This process does not therefore provide a means of meeting strict automotive  
industry standards.

## SUMMARY OF THE INVENTION

Accordingly, it is an object of the invention to provide a coating process  
which provides good adhesion values and layers of homogeneous thickness,  
25 especially thin layers (120-150  $\mu\text{m}$ ).

## BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 shows a schematic diagram of the pipe coating process where

- 1 Cleaning system
- 2 Drive 1

- 3      Primer unit
- 4      Induction (I) (coil I – primer drying)
- 5      Induction (II) (coil V – primer drying)
- 6      Radial fan 1
- 5      7      Radial fan 2
- 8      Drive 2
- 9      Induction (III) (preheating)
- 10     Lay-on roller 1
- 11     Fluidized-bed sinter basin inc. induction (IV)
- 10     12     Drive 3
- 13     PIPE
- 14     Blow-off nozzle
- 15     Lay-on roller 2
- 16     Water bath
- 15     17     Lay-on roller 3
- 18     Lay-on roller 4
- 19     Drive 4
- 20     Drive 5 (caterpillar draw-off)
- 21     Take-off

## 20                      DETAILED DESCRIPTION OF THE INVENTION

A more complete appreciation of the invention and many of the attendant advantages thereof will be readily obtained as the same becomes better understood by reference to the following detailed description when considered in connection with the accompanying drawing.

25                      The system preferably operates automatically and continuously to externally coat pipes by fluidized-bed coating. The process may also coat any pipe surface including internal, external or both. The process includes:

- 1)      a pretreatment system for cleaning the pipes, which may be internally and/or externally contaminated with or coated with grease;
- 30      2)      a primer-adhesion promoter basin for applying the adhesion promoter between steel surface and polymer layer (spray system or immersion system);

- 3) a medium-frequency induction coil (I) for baking the primer and, if a solvent-containing primer is used, for evaporating the solvent;
- 4) a radial fan for faster dissipation of the evaporated solvent;
- 5) a medium-frequency induction coil (II) for preheating the pipe;
- 5 6) a fluidized-bed coating basin with integrated medium-frequency induction coil (III) for applying the coating material. The dissipation factor of the coating material is too low for it to become heated, whereas the preheated steel pipe passing through the system is heated very rapidly to the desired temperature. The preheat temperature and immersion time provides a means to control coating thickness during fluidized-bed coating. When a pipe is passed through the process, the coating layer thickness can be changed via the generator power and the advance rate of the pipe. Preheat temperature and immersion time can be controlled independently of one another from a control desk;
- 10
- 15 7) the fluidized-bed coating basin includes an air-flush system above the pipe for eliminating powder accumulations and metal flow-guide panels below the pipe for eliminating powder deficits and any resultant pores on the underside of the pipe. Pipes having uniform layer thickness, both radially and axially, can be reliably produced when the fluidized-bed coating basin contains such devices;
- 20
- 8) a medium-frequency induction coil (IV) for smoothing the incompletely molten coating;
- 9) a melting section, needed for thorough melting of the adherent coating deposit after the pipe emerges from the medium-frequency induction coil (IV), and for producing a smooth melt. During passage through the process, the layer of adhered material is still hot and soft and is therefore easily damaged. Passing the pipe over rollers in this phase is not permissible;
- 25
- 10) an air-flush system for preliminary cooling of the pipe surface. The pipe surface temperature is thereby reduced to below the melting point of the coating material;
- 30
- 11) a water-based cooling system. The pipe runs into a water trough in which the coating layer undergoes further cooling and hardening. After cooling it is again possible to contact the pipe with rollers.

Depending on the desired layer thickness, the induction coils (II), (III) and (IV) may be operated in various combinations and with varying power including (II) and (IV),

(II) and (III),

5 (II), (III) and (IV),

(III),

(III) and (IV).

In all cases, the pipes are heated by medium-frequency induction.

Formulae for the electrical energy required, the coating rate of the process, and the  
10 powder usage are provided herein. Using the process it is possible to couple together pipe sections of any desired length to give a continuous line, which is externally coated with polymer powder as it passes horizontally through the process. Suitable coating materials are fusible polymers capable of fluidization, and mixtures of these. Polyamide powders are particularly suitable, especially  
15 those based on nylon-11 or nylon-12. Powders which give particularly good processing are described in DE 29 06 647 (Hüls AG), the trade name for which is VESTOSINT (Degussa AG). These powders have a particularly round granular shape due to preparation by a precipitation process.

A commercially available adhesion promoter (e.g. VESTOSINT adhesion  
20 promoter WS 5) is first applied to the pipe surface. Suitable primers here include any of the familiar grades for polymers, in particular those for polyamides. They may be applied in solution, suspension, or in powder form. Particularly suitable adhesion promoters for VESTOSINT are those specifically adapted to VESTOSINT. The layer thickness of the primer after air-drying is typically from  
25 5 to 8  $\mu\text{m}$ . If a solvent-containing adhesion promoter is used the solids content is generally about 8%. If use is made of a solvent-containing adhesion promoter whose solids content is about 8%, the layer thickness of the primer after air-drying is from 5 to 8  $\mu\text{m}$ . The process of the invention can achieve uniform layer thicknesses of from 50 to 1000  $\mu\text{m}$ . Preferred coating layer thicknesses are from  
30 50 to 300  $\mu\text{m}$ . The process of the invention can achieve tolerances of  $\pm 30\%$  in the coating layer thickness.

Pipes produced by the process of the invention are particularly suitable as hydraulic piping and brake piping, e.g. for the automotive industry.

Medium-frequency induction heating provides a heating method which is readily controllable but nevertheless very fast, and gives the further advantage that the induction coil which heats the pipe as it passes through the system can be placed directly within the fluidized powder, with the result that there are no heat losses. At 10,000 Hz and with a pipe wall thickness of 2 mm, heating by 300°C takes 1 s. At lower frequencies the greater penetration depth makes the heating process even faster, and at 2000 Hz the time would be only 0.73 s under the same conditions.

The induction coil takes the form of coiled tubing and is cooled by passage of water. The induction coil remains cold, as does the powder. The generator system is composed of a machine generator which generates the high frequency, a control cabinet with a control desk, a capacitor battery, and an induction coil. The system may simply be regarded as a transformer with high-frequency electrical energy fed into its primary side and with the workpiece forming its secondary side composed of just one winding. The resultant very high current density in the secondary circuit results in fast heating. Workpieces passing through the system preferably have uniform cross section and uniform wall thickness, including articles with rotational symmetry, such as wires, pipes, rods, and the like.

The throughput rate (advance rate) of the pipes depends on pipe diameter and wall thickness, i.e. on the weight of the pipe per unit length, and also on the generator power. The generator efficiency and the required degree of heating of the pipe also play a role in throughput rate. However, since these latter variables can initially be regarded as constant or at least not subject to great variation, appropriate average numerical values can be assumed. The generator power required is:

$$N = G \cdot c_p \cdot \frac{\Delta t}{860\eta} \quad (1)$$

where N is the generator power in kW, G is the pipe weight in kg/h passing through the system,  $c_p$  is the specific heat of steel ( $\sim 0.12$  kcal/kg degree),  $\Delta t$  is the

required pipe temperature increase, and  $\eta$  is the overall efficiency of the generator system (approximately from 0.6 to 0.75). The coefficient 860 derives from the conversion 1 kW = 860 kcal/h. If Eq. (1) is solved for  $G$  with  $\eta = 0.7$  and  $\Delta t \sim 240^\circ\text{C}$  the result is a very useful rule-of-thumb formula for the maximum amount of steel in kg/h which can be heated with a particular generator power (valid for the present conditions and similar conditions). This value may be greater or smaller depending on the overall process conditions.

$$G \approx 20 N \quad (2)$$

Using a 36 kW medium-frequency generator, therefore, about 720 kg/h of steel pipe can be heated by  $240^\circ\text{C}$ .

For quick approximations, an approximate guideline value for power required (under the present conditions or similar conditions) is:

$$N \approx 50 \frac{W}{\text{kg} / \text{h}} \quad (3)$$

These rule-of-thumb formulae are naturally not dimensionally correct since the numerical value has been used for quantities which have dimensions (e.g. specific heat  $c_p$ ). However, these dimensionally incorrect equations have proven to be very useful for operating requirements. If the formulae for the weight of pipe passing through the system are combined with the formula for the generator power required, the result is a simple relationship for the maximum throughput rate (advance rate) of a steel pipe of density  $\gamma = 7.85 \text{ kg/dm}^3$  for a given generator power. If, for example, there is a frequent need for pipes of various diameters and wall thicknesses to be coated in a pipe-coating system, the following formula rapidly gives a guideline value for the maximum throughput rate. The numerical factor has to be altered somewhat for other conditions. The actual throughput may be greater or smaller depending on the overall process conditions:

$$v_{\max} \approx 18 \frac{N}{(d_a - s) \cdot s} \quad (4).$$

The throughput rate  $v_{\max}$  has to be inserted here in m/min, the generator power  $N$  in kW, the external pipe diameter  $d_e$  in mm, and the pipe wall thickness  $s$  likewise in mm.

5 Example:

A pipe-coating process was used to prepare coated pipes. The process includes:

- 1) a pretreatment system for cleaning the pipes, which are mostly supplied greased;
- 10 2) a primer-adhesion promoter basin for applying the adhesion promoter between steel surface and polymer layer (spray system or immersion system);
- 3) a medium-frequency induction coil (I) for baking the primer and for evaporating the solvent;
- 4) a radial fan for faster dissipation of the evaporated solvent;
- 15 5) a medium-frequency induction coil (II) for preheating the pipe;
- 6) a fluidized-bed coating basin with integrated medium-frequency induction coil (III) for applying nylon-12. The dissipation factor of the polyamide powder is too low for it to become heated, whereas the preheated steel pipe passing through the system is heated very rapidly to the desired temperature.
- 20 The preheat temperature and immersion time control the coating layer thickness during fluidized-bed coating. In the case of a pipe passing through the system this means that the layer thickness can be changed via the generator power and the advance rate of the pipe. The two factors can be controlled independently of one another from a control desk;
- 25 7) the fluidized-bed coating basin includes an air-flush system above the pipe for eliminating powder accumulations and metal flow-guide panels below the pipe for eliminating powder deficits and the resultant pores on the underside of the pipe. Uniform coating layer thickness, both radially and axially, can be ensured only by using this fluidized-bed coating basin;

- 8) a medium-frequency induction coil (IV) for smoothing the incompletely molten coating;
- 9) a melting section, for thorough melting of the adherent coating deposit after the pipe emerges from the medium-frequency induction coil (IV), and for  
5 producing a smooth melt. During passage through the system, the layer is still hot and soft and is therefore easily damaged. Passing of the pipe over rollers in this phase is therefore not permissible;
- 10) an air-flush system for preliminary cooling of the pipe surface. The pipe surface temperature is thus cooled below the melting point of the coating  
10 material;
- 11) a water-based cooling system. The pipe runs into a water trough in which the coating layer undergoes further cooling and hardening, after which guiding over rollers becomes possible again.

The results of a series of experiments on the system described are given in  
15 Table 1. All of Examples 1 to 7 used VESTOSINT 2157 precipitated nylon-12 powder from Degussa AG. In all the examples given there was no chromate-pretreatment.



### Experiments on continuous pipe-coating using PA 12

Examp le	Experiment No.	Ø pipe [mm]	V <sub>pipe</sub> [m/min]	Coil					Powder level (without fluidiz- ation) [mm]	Internals in fluidized-bed pan		Layer [µm]	Remarks
				I [kW]	II [kW]	III [kW]	IV [kW]	V [kW]		Gap [mm]	Nozzles [bar]		
1	980420- 001	12x1	14	-	10	1.5	4.5	-	160	7	2-1-1-2	130-160	
2	980421- 003	12x1	14	-	10	1.5	4.5	-	165	7	2-1-1-2	120-160	
3	980422- 003	12x1	14	-	9	1.5	7.5	-	163	7	2-1-1-2	100-140	
4	980422- 010	12x1	14	11.2	1.5	1.5	7.5	-	160	7	2-1-1-2	120-140	Primer
5	000619-1	10x1	5.5	2.4	2.7	3.6	4.86	5.2	160	6	2-1-1-2	100-160	Primer
6	000620-3	10x1	5.5	2.4	2.7	3.6	4.86	5.2	163	8	2-1-1-2	120-160	
7	000620-4	10x1	5.5	2.4	2.7	3.6	4.86	5.2	163	4	2-1-1-2	80-160	Primer

Coil I Primer drying I

Coil II Pipe preheating

Coil III Fluidized-bed coating pan

Coil IV Smoothing

Coil V Primer drying V

### Tests on primed pipes

- a) TL 222 corrosion-protection coatings on brake pipes (surface-protection requirements) **D Zn/PA**

Corrosion resistance: test time 500 h with scribe mark to DIN 53 167;

5 Scribe mark creep  $\leq 2\text{mm}$

Corrosion resistance: test time 500 h following rock impact test to PV 1213; no underlying metal corrosion

Corrosion resistance: test time 1000 h; no zinc corrosion, no underlying metal corrosion, and no breakaway of polyamide layer

10 Chemicals resistance: to TL222, item 5; no blistering or softening of the polymer layer occurred after 24 hours of air-drying and then winding around a mandrel (360°) of dimension 16 mm, no visible cracks or peeling of the polyamide coating occurred

- b) Adhesion tests on primed pipes after storage in water, tests using knife tip:

15 Pipes without scribe mark

Dry test, one day after coating: very good adhesion

Dry test, one day after coating, on a wound pipe (around 16 mm mandrel); very good adhesion

3 days' storage in water, directly after removal, very good adhesion

20 Pipes with scribe mark

Dry test, one day after coating: very good adhesion

3 days' storage in water, directly after removal, very good adhesion

Obviously, numerous modifications and variations of the present invention are possible in light of the above teachings. It is therefore to be understood that within the scope

of the appended claims, the invention may be practiced otherwise than as specifically described herein.

German Application 1023345.9 filed on July 23, 2002 is incorporated herein by  
5 reference in its entirety.